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Research Paper

Performance of $CaCl_2$-reactor for application in ammonia-salt based thermal transformers

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ABSTRACT

Thermochemical reactions, such as calcium chloride reacting with ammonia to form calcium chloride complexes, are attractive for application in heat pumps as they produce more heat per kg adsorbed sorbate but also adsorb a considerable higher amount of sorbate per kg of sorbent, compared to adsorbents such as zeolites. These benefits, however, come with together with a number of challenges. Firstly, the salts have poor thermal conductivity so the path from heat exchanger to the sorbent must be limited in order to allow for short cycling times and therefore high power density. Secondly, the salts typically swell and shrink upon (de)sorption, thereby easily losing their contact with the heat exchanger. To overcome these two problems, the salts are often placed in a matrix, such as expanded natural graphite (ENG) or zeolites.

This paper shows the performance of a reactor containing approximately 1 kg of $CaCl_2$ placed in a 1 kg ENG matrix. Its performance in terms of adsorption/desorption rates, heat input and output as a function of temperature and pressure gradients and under typical heat pump and transformer conditions is shown and compared with model calculations. The parameters used in the model calculations have been obtained from literature or independently measured using apparatus such as Rubotherm microbalance and a large temperature jump setup. The results show that material properties measured on small samples do not easily yield a proper description of the sorbent performance on kW-scale reactors. Some suggestions are made to improve future model description and experiments.

1. Introduction

Thermally driven heat pumps and transformers can contribute significantly to reducing our carbon footprint. Various types of thermally driven heat pumps and transformers can be found in literature, each with their own benefits and challenges. In this paper we focus on the development of thermally driven system using thermochemical reactions. These are reactions between a sorbate (e.g. ammonia) and a sorbent (e.g. $CaCl_2$) in which heat is released when combined and heat is extracted when separated.

Although a considerable amount of research has been done [1–4], few or no commercial thermochemical systems are currently available. The main challenge is to achieve stable performance with sufficiently high power density to allow for reasonably compact and affordable systems. In the past, the Energy Research Centre of the Netherlands (ECN) has built several prototypes using various heat exchanger designs but each had their own problems preventing further development to a commercial product. After these setbacks, it was decided to develop a unit in which reactor elements could be tested before building an entire reactor and system.

The work presented in this paper takes one of the concepts that was successfully tested in the aforementioned setup and placed it inside a 10 kW scale reactor for testing. It describes the performance of a reactor based on $CaCl_2$ impregnated in ENG. The measurements are subsequently compared to model calculations using thermal conductivity and reaction kinetics data from separate measurements or literature sources.

Fig. 1 shows earlier prototypes of thermochemical heat transformers developed at ECN. The first prototype (left) contained finned tube heat exchanger with a packed bed of salt. Probably due to swelling of the salt the ammonia could not be cycled at a sufficient rate. A second prototype was built (middle) where salt was deposited in a metal-foam placed on a sandwich plate through which thermal oil was flowing. This system had two major problems: the first one was the enormous thermal mass of the system,

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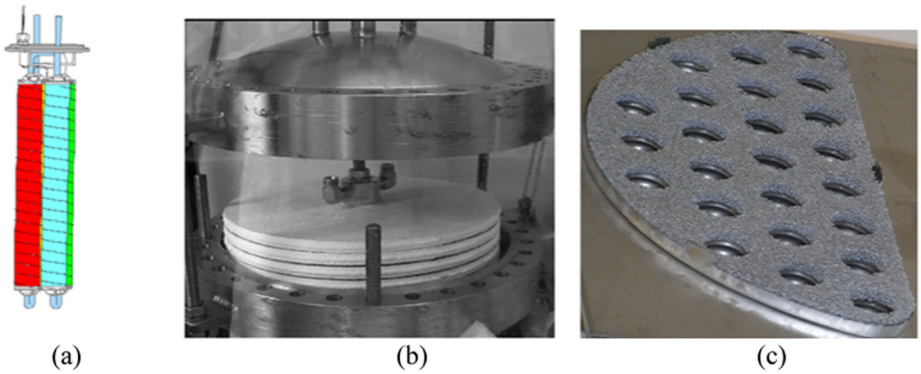

Fig. 1. Earlier thermochemical heat transformer prototypes developed and tested at ECN [5–7]. Left (a) was based on a finned tube heat exchanger design, middle (b) used a brazed plate configuration with Ni-foam as salt-matrix, right (c) shows part of a shell-n-tube heat exchanger design with heat exchanging fluid inside the tubes and aluminium foam is placed in aluminium container around the tubes to hold the salt in place.

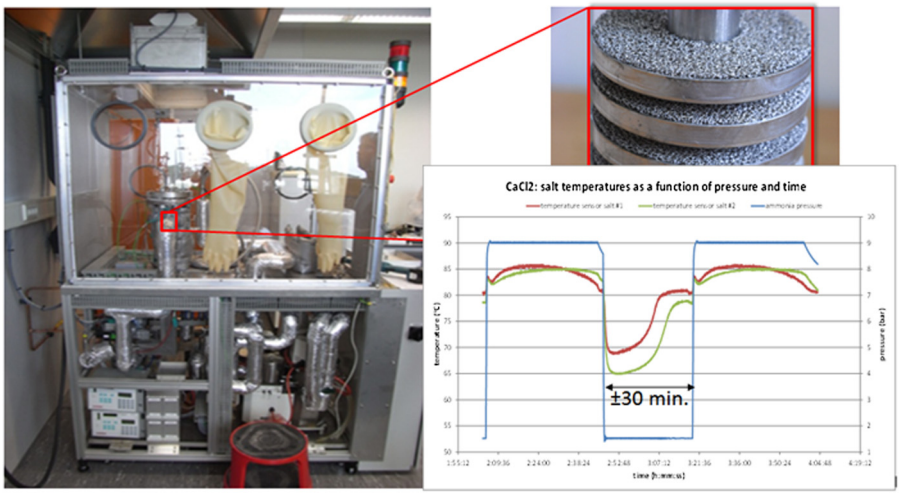

Fig. 2. Lab-scale setup (left), reactor element (top right) and result $CaCl_2(4\text{-}8)NH_3$ (bottom right).

both in the sandwich plates and the flanges/domes and secondly the ammonia uptake was (too) slow, possibly because of too high loading of salts in the metal foam.

A third prototype (right) was developed. This time thermal mass was reduced, using a shell-and-tube type heat exchanger with thermal oil on the tube side and plates with aluminium foam on the shell side.

Although the reactors contained about 2.5 and 1.3 kg of salts, LiCl and $MgCl_2$ respectively, and a volume of several litres, measured power peaked at around 500 W. Analysis were unclear about what caused the low power density although recent experiments showed lithium chloride ammonia complex melts at 5 bar ammonia pressure and temperatures above 95 °C. This could explain the poor performance as it is the operating conditions of the heat transformer. Also poor thermal heat transfer from the thermal oil to the tubes and fins could have contributed to the poor performance.

In order to allow for more rapid development of heat exchanger types, salt loading methods and cyclic stability, the lab-scale setup has been constructed, see Fig. 2 [8]. This setup allows for testing heat exchanger elements rather than having to build entire systems. Results using a similar aluminium foam as used in the previous heat transformer system in combination with calcium chloride showed repeatable results but charging/discharging times still took 30–60 min, resulting in too low power densities for industrial application.

After these experiments, the concept of a fin containing an aluminium foam with salt was abandoned and replaced by ENG in tube concept with oil/water on the shell side. The results for $MgCl_2$ in ENG disks is shown in Fig. 3 [9]. Similar results are obtained for measurements on LiCl, $MnCl_2$, $CaCl_2$ and $ZnCl_2$. The measurements also showed good cyclic stability for more than 1000 cycles. With half cycle times of less than 600 s, application in industrial systems becomes feasible.

2. Experimental setup

At the heart of the experimental setup is a carbon steel shell-and-tube reactor containing the ENG-$CaCl_2$ matrix inside of the eight, 1.2 m long tubes and thermal oil as the heat transfer fluid on the shell side (see Table 1 for details). The 1000 ENG disks that form the ENG-$CaCl_2$ matrix have made by cutting them from a SGL Thermophit plate with a density of 150 kg m^{-3}. The disks were weighted and subsequently put in a container with ethanol. Remaining air was removed from the ENG disks by evacuating the container. Subsequently the disks were placed in a container with a $CaCl_2$-solution of 375 g $CaCl_2$ per litre water. The disks were removed from the $CaCl_2$-solution after 48 h and placed in an oven for 24 h at 90 °C followed by 24 h in the oven at 200 °C to remove all water. The disks impregnated with $CaCl_2$ were then weighed to determine the amount of $CaCl_2$ in the disks and subsequently

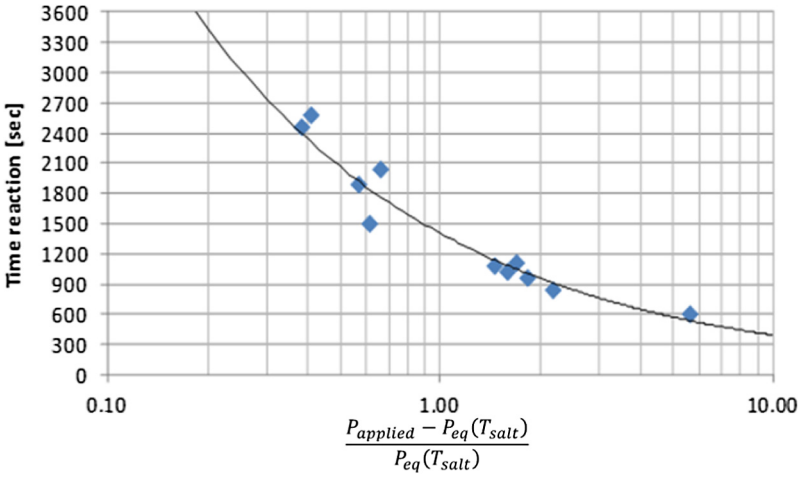

Fig. 3. Small-scale lab setup results for reaction time as a function of relative pressure ratio defined as $P_{applied}/P_{eq}(T_{salt}) - 1$, for 10 g $MgCl_2$ in ENG [9]. The measurements have been conducted for a range of temperatures (120–180 °C) and pressures (3–8 bar). The reaction time has been defined as the amount of time required to reach 90% of the total amount ammonia adsorbed for $MgCl_2$(2-6)NH_3 reaction.

Table 1
Reactor and sorbent properties.

Property	Value
Weight ENG	870 g
Weight $CaCl_2$	927 g
Inner diameter tubes	28 mm
Outer diameter tubes	31.6 mm (1¼″)
Diameter shell	152 mm (6″)
Volume tube-side	5.9 dm^3
Volume shell-side	11.2 dm^3
Length tube	1.2 m per tube
Number of tubes	8
Heat transfer fluid	Oil
Connection shell side	18.9 mm (¾″)
Connection tube side	12.1 mm (½″)

placed in the reactor whilst minimizing exposure to ambient air to avoid hydration of the calcium chloride.

The reactor is connected to the so-called ThermExS facility that supplies or extract heat as shown in Fig. 4. The ammonia is supplied using a shell-and-tube type evaporator, and removed using a plate heat exchanger type condenser, both capable of providing 10+ kW evaporator/condenser power. The reactor, condenser and evaporator can be set to a desired temperature using Huber thermostatic baths. The flow of ammonia is controlled by opening the valves between the reactor and the evaporator and condenser. Typically the reactor is varied between two temperatures (and pressures) while the evaporator and condenser are kept at a constant temperature and pressure. The latter allows for accurate measurement of heat fluxes to/from the condenser and evaporator. For the equilibrium measurements the valves between the reactor and the evaporator/condenser were closed to maintain the state of the calcium chloride ammonia complex.

The measured parameters include mass flow of the thermal oil, water and glycol, the temperatures of fluid entering and leaving the reactor, evaporator and condenser and the ammonia pressures in these components. Also a temperature sensor has been inserted in the ENG disks to measure the temperature of the salt-ENG composite. The pressure measurement is accurate to 0.5% F.S. and the thermocouples are within 0.1 K accurate compared in relation to the reference temperature. Heat flows to/from the different components are calculated using the mass flows, heat capacity and temperatures. Pressure is measured in the reactor and the condenser and evaporator.

3. Results

3.1. Equilibrium measurements

Fig. 5 shows the result of the measurement of the (quasi) equilibrium between pressure and temperature by increasing and subsequently decreasing the reactor temperature in steps of 10 K whilst the connections to evaporator and condenser remained closed. The right graph shows the measured pressure – temperature relation in comparison with the theoretical correlation based on the Claussius-Clapeyron equation with the ΔS and ΔH data for the reactions as shown in Table 2. As can be seen from the right graph, the measured data (blue line) correlates very well with the theoretical P-T relation for the $CaCl_2$(4-8)NH_3 reaction (green line) based on the measured salt temperature (T_{salt}). This means hysteresis is negligible and that the thermocouple placed in the ENG-salt composite yields a representative value for the salt temperature. For the $CaCl_2$(4-2)NH_3 reaction (not shown) a minor deviation (<5 K) is found, likely due to (slow) reaction kinetics.

Claussius-Clapeyron equation:

$$P = P_0 e^{-\frac{\Delta H - T\Delta S}{RT}} \quad (1)$$

3.2. Dynamic measurements

Fig. 6 shows an example of a dynamic measurement of the desorption of ammonia from the $CaCl_2$-ENG composite by heating the reactor from 40 °C to 130 °C with a condenser temperature of 40 °C (16 bar NH_3) and a evaporator temperature of −10 °C (3 bar NH_3). The blue[1] line shows the salt temperature, which increases from 40 °C to 130 °C. The green and red lines show the equilibrium temperatures for respectively the $CaCl_2$(8-4)NH_3 and the $CaCl_2$(4-2) NH_3 reaction based on the pressure in the reactor. The purple line shows the power measured on the condenser (Q_{cond}), calculated from the difference in temperature in/out of the condenser and the flowrate of the water.

A rapid increase in salt temperature can be observed between 500 and 1000 s. The salt temperature remains near the temperature associated with the equilibrium condition for the desorption reaction of $CaCl_2$(8-4)NH_3 for the measured ammonia pressure

[1] For interpretation of color in Fig. 6, the reader is referred to the web version of this article.

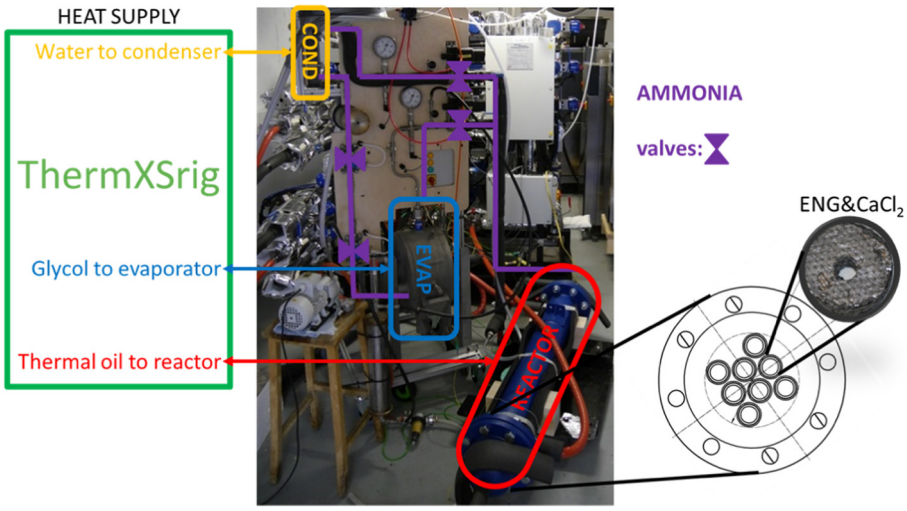

Fig. 4. Reactor filled with ENG-$CaCl_2$ disks connected to ThermExSrig facility for supplying/extracting heat and ammonia. The evaporator is connected to a glycol loop, the condenser to a water loop and the reactor to a thermal oil loop.

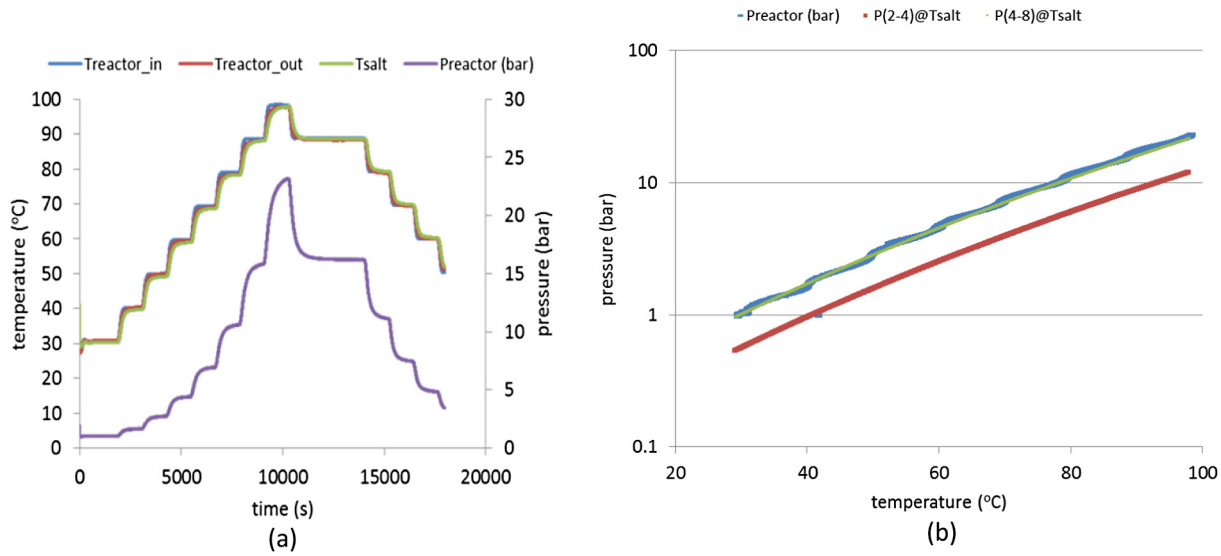

Fig. 5. Left (a) shows on the left-axis the measured temperatures for respectively oil entering/leaving the reactor and ENG-$CaCl_2$ in reactor (blue, red, green lines) and on the right axis the measured ammonia pressure (purple) in the reactor as a function of time. Right (b) shows the measured ammonia pressure (blue line) as a function of $CaCl_2$-ENG temperature together with the theoretical pressure-temperature relation for $CaCl_2$(2-4)NH_3 (red line) and for $CaCl_2$(4-8)NH_3 (green line) based on Claussius-Clapeyron Eq. (1). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Table 2
Entropy and enthalpy values for $CaCl_2$(2-4)NH_3 and $CaCl_2$(4-8)NH_3 reactions [10]

	$CaCl_2$(2-4)NH_3	$CaCl_2$(4-8)NH_3
ΔH (J mol^{-1})	42100	42100
ΔS (J mol^{-1} K^{-1})	134.1	139.0

(~16 bar) whilst the condenser power shows peak power of 1.6 kW. The salt temperature rises further to about 110 °C for another 500 s after which the condenser power returns to zero, indicating the desorption reaction has finished. The salt temperature rises further until it reaches the (set) temperature of the thermal oil (130 °C). At t = 3000 s the reactor and thereby the salt is cooled down again to start adsorption of ammonia again. The temperature profile and the condenser power both indicate a two-step reaction, suggesting a first desorption step of $CaCl_2$(8-4)NH_3 and a second desorption step of $CaCl_2$(4-2)NH_3. After the second desorption the temperature of the reactor and the salt increases further till it reached the temperature of the thermal oil (130 °C).

3.3. Model calculations

Dynamic measurements have been modelled using a 1-D Matlab model that includes heat transfer resistance from reactor wall to the ENG-$CaCl_2$ composite, thermal conductivity through the composite and kinetic behaviour of the sorbent. The input parameters are shown in Table 3 and have been obtained from independent measurements or literature sources. The following main equations have been used to model the sorption process.Heat transfer from thermal oil to composite material:

$$\frac{dQ}{dt} = U \cdot A_{tube}(T_{oil} - T_{wall}) \quad (2)$$

Heat transfer & production at position *i* in composite material:

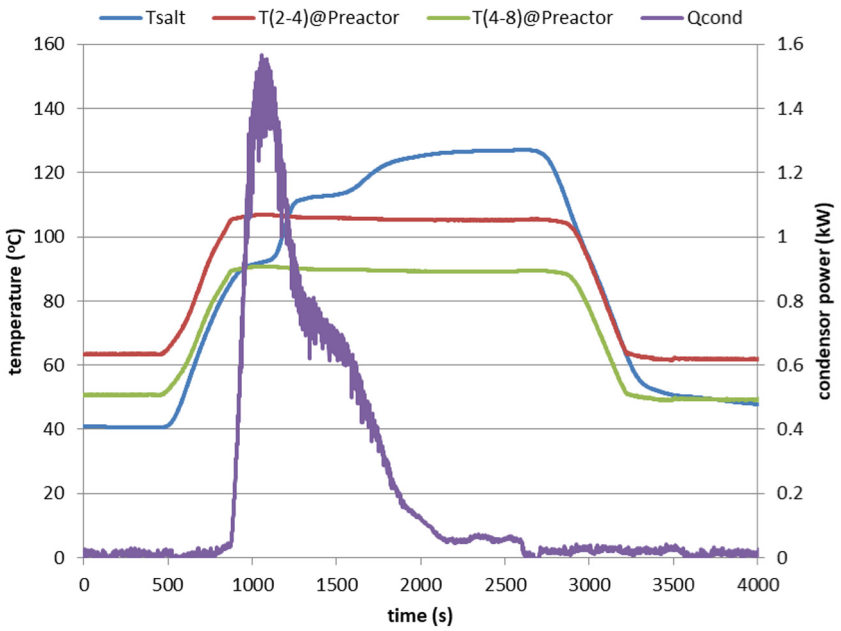

Fig. 6. Dynamic measurement of salt temperature (T_{salt}) and condenser power (Q_{cond}) as a function of time. T(2-4)@Preactor and T(4-8)@Preactor are the temperatures associated for the reactor pressure in equilibrium for respectively $CaCl_2$(2-4)NH and $CaCl_2$(4-8)NH_3 reaction and has been calculated by using a rearranged version of Eq. (1): $T = \frac{\Delta H}{\Delta S - R \ln \frac{P}{P_0}}$.

Table 3
Main input parameters for the Matlab model calculations.

Parameter	Value	Source
Pressure in reactor	9 bar	Experimental conditions
Initial temperature (t = 0)	40 °C	
Desorption temperature (t > 0)	130 °C	
Thermal conductivity ENG	6 W m^{-1} K^{-1}	Data SGL Carbon
Heat transfer coefficient oil to ENG	50/100/200 W m^{-2} K^{-1}	Estimated range according to literature
Kinetic rate constant $CaCl_2$(8-4)NH_3	0.04 s^{-1}	[11]
Kinetic rate constant $CaCl_2$(4-2)NH_3	0.04 s^{-1}	[11]

$$\frac{dQ_i}{dt} = -\lambda A_i \frac{dT_i}{dx} + \Delta H_{84} \cdot \frac{dn_{84}}{dt} + \Delta H_{42} \cdot \frac{dn_{42}}{dt} \quad (3)$$

Reaction rates at position i in composite material:

$$\frac{dn_{84}}{dt} = k_{84} \cdot \frac{P_{84}(T_i) - P_{reactor}}{P_{reactor}} \quad (4)$$

$$\frac{dn_{42}}{dt} = k_{42} \cdot \frac{P_{42}(T_i) - P_{reactor}}{P_{reactor}} \quad (5)$$

Temperature change at position i in composite material:

$$\frac{dT_i}{dt} = \frac{1}{A_i \cdot dx \cdot \rho \cdot C_p} \cdot \frac{dQ_i}{dt} \quad (6)$$

where:

A_{tube}	surface area of inner tube in contact with composite material (m^2)
A_i	surface area of composite at position i perpendicular to heat flow (m^2)
C_p	heat capacity of composite material (J g^{-1} K^{-1})
dx	length of element in direction of heat flow (m)
k_{84}	kinetic constant reaction $CaCl_2$(8-4)NH_3 (s^{-1})
k_{42}	kinetic constant reaction $CaCl_2$(4-2)NH_3 (s^{-1})
n_{84}	fraction of reaction $CaCl_2$(8-4)NH_3 completed (value from 0 to 1)
n_{42}	fraction of reaction $CaCl_2$(4-2)NH_3 completed (value from 0 to 1)
$P_{reactor}$	ammonia pressure in reactor (bar)
$P_{84}(T_i)$	equilibrium pressure for $CaCl_2$(8-4)NH_3 reaction at temperature T_i (bar)
$P_{42}(T_i)$	equilibrium pressure for $CaCl_2$(4-2)NH_3 reaction at temperature T_i (bar)
Q	heat transferred from tube to composite material (J)
Q_i	heat at position i in composite material (J)
t	time (s)
T_{oil}	temperature of thermal oil (K)
T_i	temperature of composite material at position i (K)
T_{wall}	temperature of composite material at wall (K)
U	heat transfer coefficient from oil to composite material (W m^{-2} K^{-1})
ΔH_{84}	total heat of sorption for $CaCl_2$(8-4)NH_3 at position i (J)
ΔH_{42}	total heat of sorption for $CaCl_2$(4-2)NH_3 at position i (J)
λ	thermal conductivity of composite material (W m^{-1} K^{-1})
ρ	density of composite material (g m^{-3})

Fig. 7 shows the calculated amount of ammonia desorbed for a range of heat transfer coefficients together with the amount based on measured condenser power. It can be observed that the measured amount of ammonia is about 10% less than the amount that could theoretically be desorbed. It can also be seen that a heat transfer coefficient of 100 Wm^{-2} K^{-1} shows a good correlation with measured data. The temperature profiles from the model calculations have a reasonable correlation with the measured values (left

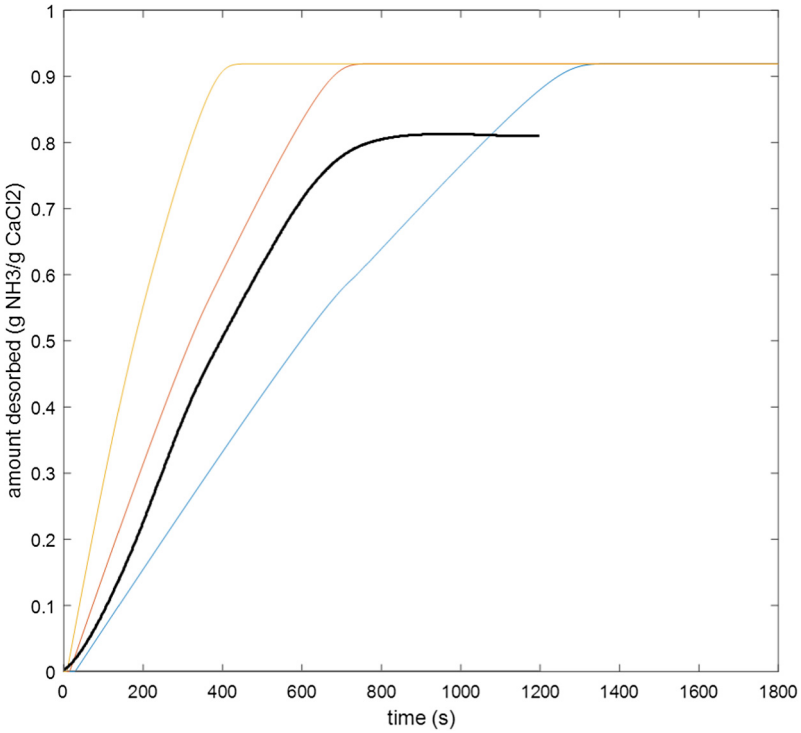

Fig. 7. Amount of desorbed ammonia (g NH_3 per g $CaCl_2$) measured value (black line) and values based on model calculations using heat transfer coefficients of respectively 50 (blue line), 100 (red line) and 200 (yellow line) $Wm^{-2}\,K^{-1}$ as a function of time. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

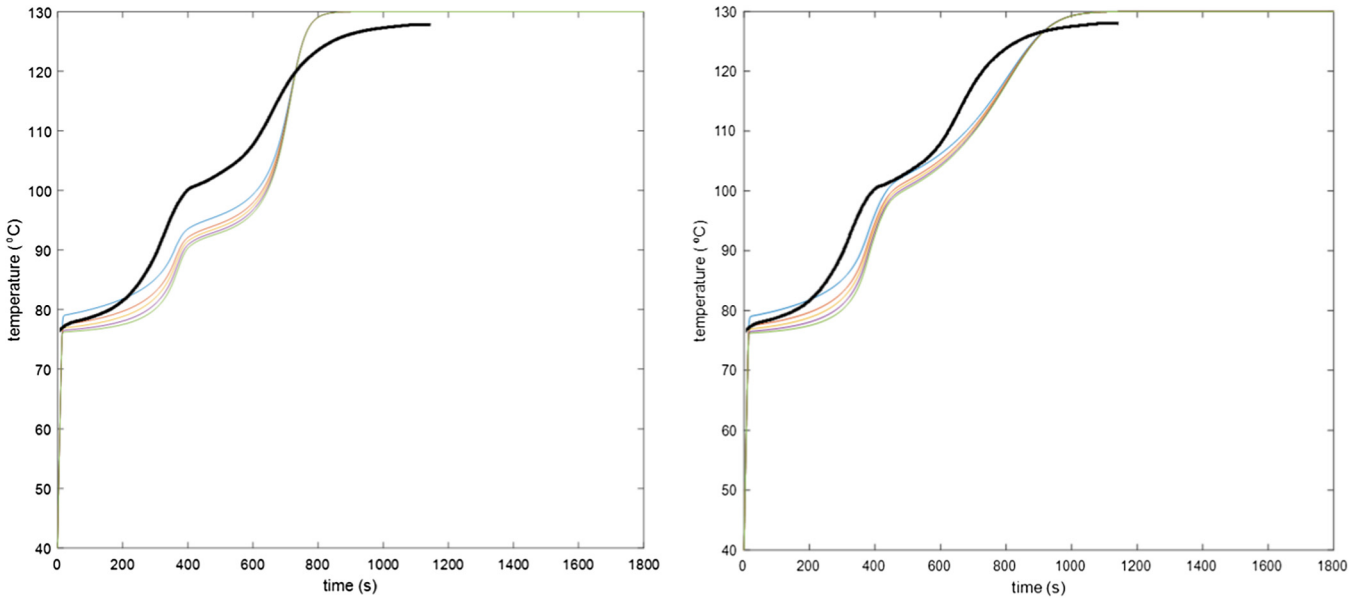

Fig. 8. Temperatures at five locations from model calculations as a function of time with original (left) and a 5-times reduced (right) reaction kinetics for the $CaCl_2$(4-2)NH_3 reaction and assuming 100 $Wm^{-2}\,K^{-1}$ heat transfer coefficient for both cases. The bold black line shows the measured temperature profile as function of time.

graph of Fig. 8) and a good correlation when a 5-times smaller kinetic rate constant is used for the $CaCl_2$(4-2)NH_3 reaction (right graph of Fig. 8).

4. Discussion

Measurements on reactor show good correlation with equilibrium conditions for the $CaCl_2$(8-4)NH_3 reaction, both adsorption and desorption. Therefore the driving force for the desorption of ammonia from $CaCl_2$ can be limited to heat transfer limitations, due to transfer of heat from thermal oil to the composite material and within the composite material itself, and limitations in reaction kinetics. Model calculations show a good correlation with measurements, both in terms of rate of uptake (Fig. 7) as well as the temperature profile measured and modelled (Fig. 8) provided a slight limitation is introduced on the reaction kinetics of the $CaCl_2$(4-2)NH_3 reaction. The latter is contradicting the large temperature jump measurements conducted on $CaCl_2$ by Jegede [2]

that showed equal kinetic rate constants for both reactions. The phenomenon could be future investigated by conducting more experiments over a larger range of ammonia pressures and salt temperatures. Given the small difference in measured temperature and equilibrium temperature for the $CaCl_2(8\text{-}4)NH_3$ reaction, improving the heat transfer rate from the heat transfer fluid to the composite material is expected to have a large positive effect on the rate of reaction and thereby reducing the cycle time significantly. This improvement can be achieved by either increasing the heat transfer surface area (per kg of sorbent) which can be done by using smaller diameter tubes and/or by replacing the thermal oil by water or steam as heat exchanging medium. A factor of 4 increase in heat transfer coefficient can be expected for the heat transfer medium. The overall effect can be smaller as the current value also includes the transfer of heat through the tube to the composite material.

Despite the limitations on heat transfer, a desorption rate of about 800 g NH_3 in 800 s has been observed. Given the typical sorption enthalpy of 2.5 kJ/g NH_3 for ammonia salts for a resorption, thermal transformer system, this is equal to 2.5 kW sorption heat for the reactor or a power density of 420 kW per m^3 sorbent volume. Efficiency of a thermal transformer based on the current reactor is very low (COP < 0.1) because about 75% of sorption heat is required for a 50 K temperature rise of the reactor itself. This, however, is mainly due to the large thermal mass of the shell, headers and the flanges that connects them. For large-scale, industrial application the thermal mass of the shell and headers compared to the sorption heat will be considerably less. Also the thermal of the heat exchanging medium can be reduced by having a more compact design of the tubes. Finally, thermal transformer can be executed as a four-bed system with heat recovery between the beds. Overall it is estimated a full-scale thermal transformer upgrading waste heat of 130–180 °C process heat can reach a COP between 0.35 and 0.40 where a COP of 0.50 is (roughly) the maximum achievable value for a resorption-based thermal transformer. Before constructing a bench-scale prototype thermal transformer, it is recommended to verify performance of the high temperature ammonia-salts in a similar reactor as presented in this paper to verify no hysteresis occurs and the performance is not limited by reaction kinetics or thermal conductivity in the salt matrix.

5. Conclusions

- $CaCl_2$ in ENG shows little to no hysteresis for the $CaCl_2(4\text{-}8)NH_3$ reaction, both in the adsorption and desorption process.
- There is a good correlation between the model calculations and measurements on desorption rate of ammonia from $CaCl_2{\cdot}8NH_3$
- Contrary to previous study, kinetic rate constants seem different for $CaCl_2(8\text{-}4)NH_3$ and $CaCl_2(4\text{-}2)NH_3$ reactions.
- Desorption rate of the reactor is limited by heat transfer from the thermal oil to the ENG-salt matrix, likely related to the poor heat transfer properties of the thermal oil, rather than reaction kinetics or thermal conductivity of the salt matrix.
- Outlook for reasonable performance of thermal transformer (COP = 0.35–0.40) when reactor is scaled to industrial size.

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